

A tropical garden scene with a pineapple sculpture, a fountain, and benches.

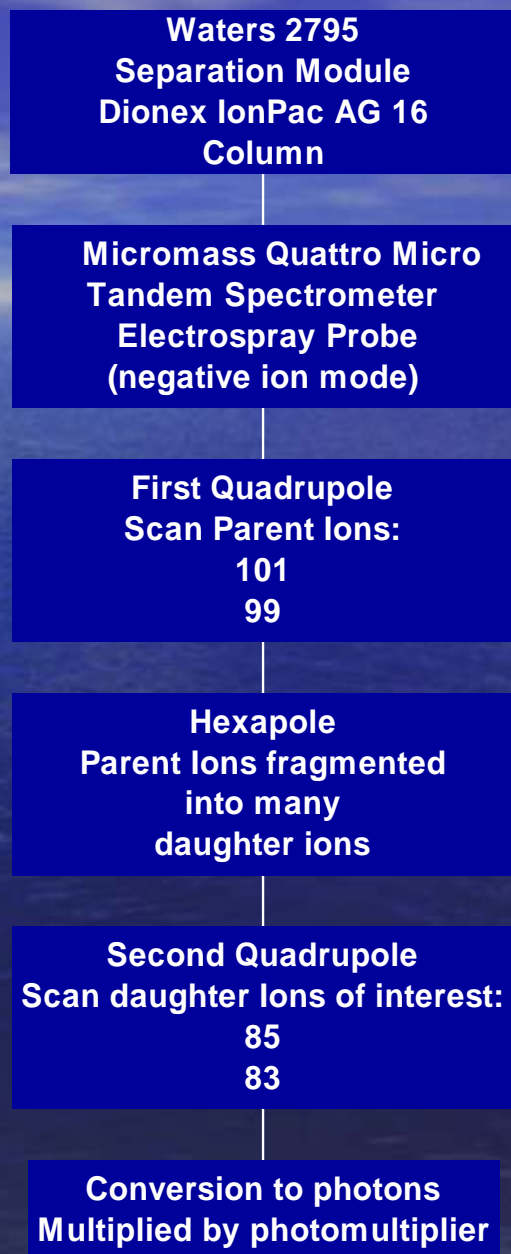
# **Low Level Perchlorate Analysis By LCMSMS**

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# Agenda

- Schematic of LCMSMS system
- Method applicability
- Method advantages
- Method cost comparision
- Quality control acceptance guidelines
- Questions

# LCMSMS Schematic





# Method Applicability

Wide Variety of Sample Matrices can be analyzed :

- Water
- Soil
- Vegetation
- Milk
- Juices
- Cleaning agents/Solvents

# Method Advantages

- Detection Limits :

Aqueous samples - 50 ppt


Solid samples - 500 ppt

- Typical matrix interferences (high organic content, chloride, sulfate, bicarbonate, and carbonate) do not greatly affect the analysis.
- Able to quantitate the perchlorate isotopic ratio (99:101).



# Method Cost Comparison

- Aqueous Samples : \$20 more than the EPA IC Method 314
- Solid Samples : \$30 more than the EPA IC Method 314



# Quality Control Acceptance Guidelines

# Perchlorate Isotopic Ratio Criteria

Study performed annually consisting of the ratio population from :

- Extracted QC (LCS, MS, MSD, ICS) from all matrices
  - Calibration standards
  - MDL verification standards
  - Calibration verification standards



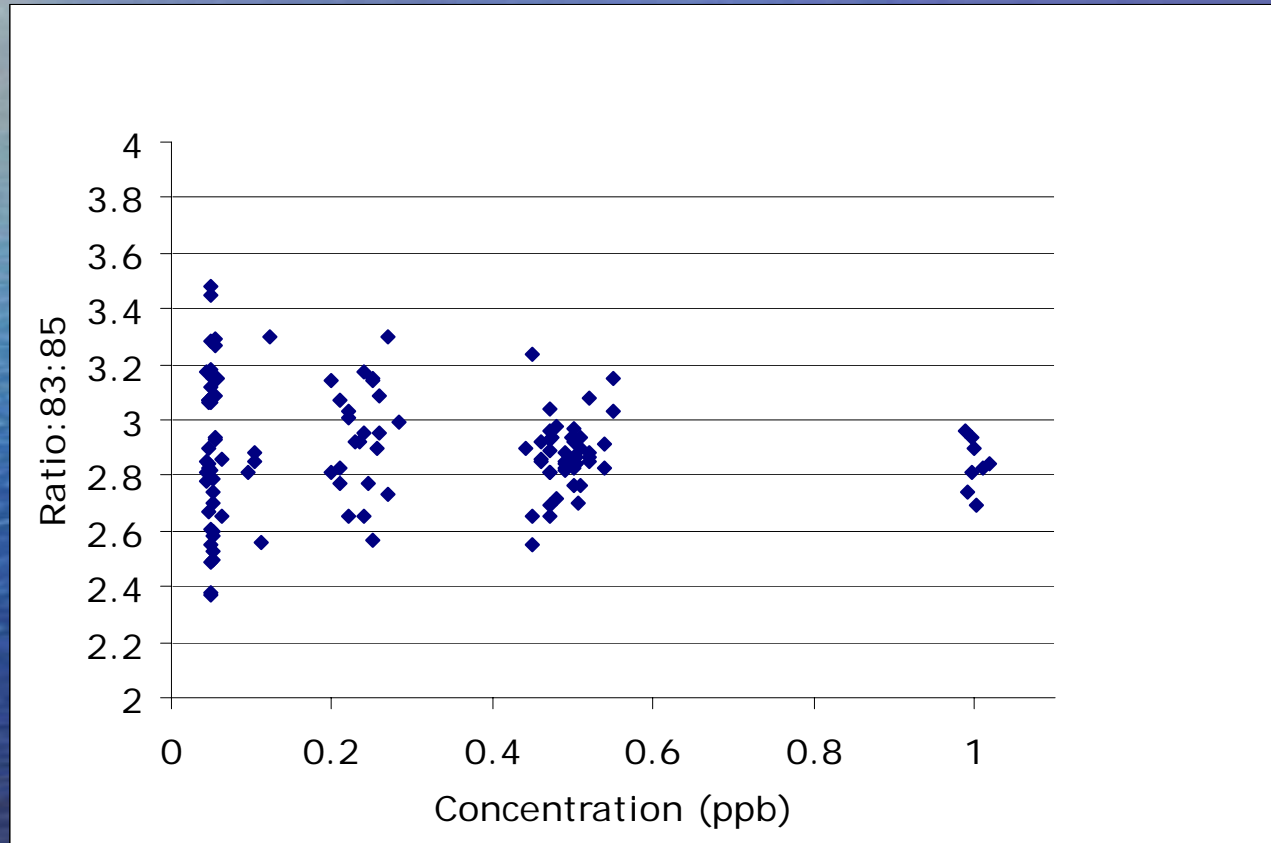
## Perchlorate Isotopic Ratio Criteria (cont.)

- Population Mean - within 10% of 3.07
- Population Standard Deviation - approximately 0.20
- For Samples whose perchlorate concentrations fall between the MDL and PQL, their ratio should be within 20% ( $3\sigma$ ) of the population mean.
- For perchlorate concentrations that are at or exceed the PQL, their ratio should be within 15% ( $2\sigma$ ) of the population mean.

## Perchlorate Isotopic Ratio Criteria (cont.)

- Mean : 2.85
- Standard Deviation : 0.20
- 15% limits : 2.67 - 3.48
- 20% limits : 2.45 - 3.69

# QC Samples and Standard Isotopic Ratios





# Calibration and Performance Criteria

- Internal Standard :
  - Perchlorate-O(18) spiked into all standards, blanks, and samples at the midpoint of the calibration level
  - Area of the standard must be within 50% of the average of the five calibration levels

# Calibration and Performance Criteria

- Daily calibration
  - Five standards
  - Linear, forced through the origin
  - Linearity coefficient  $\geq 0.995$

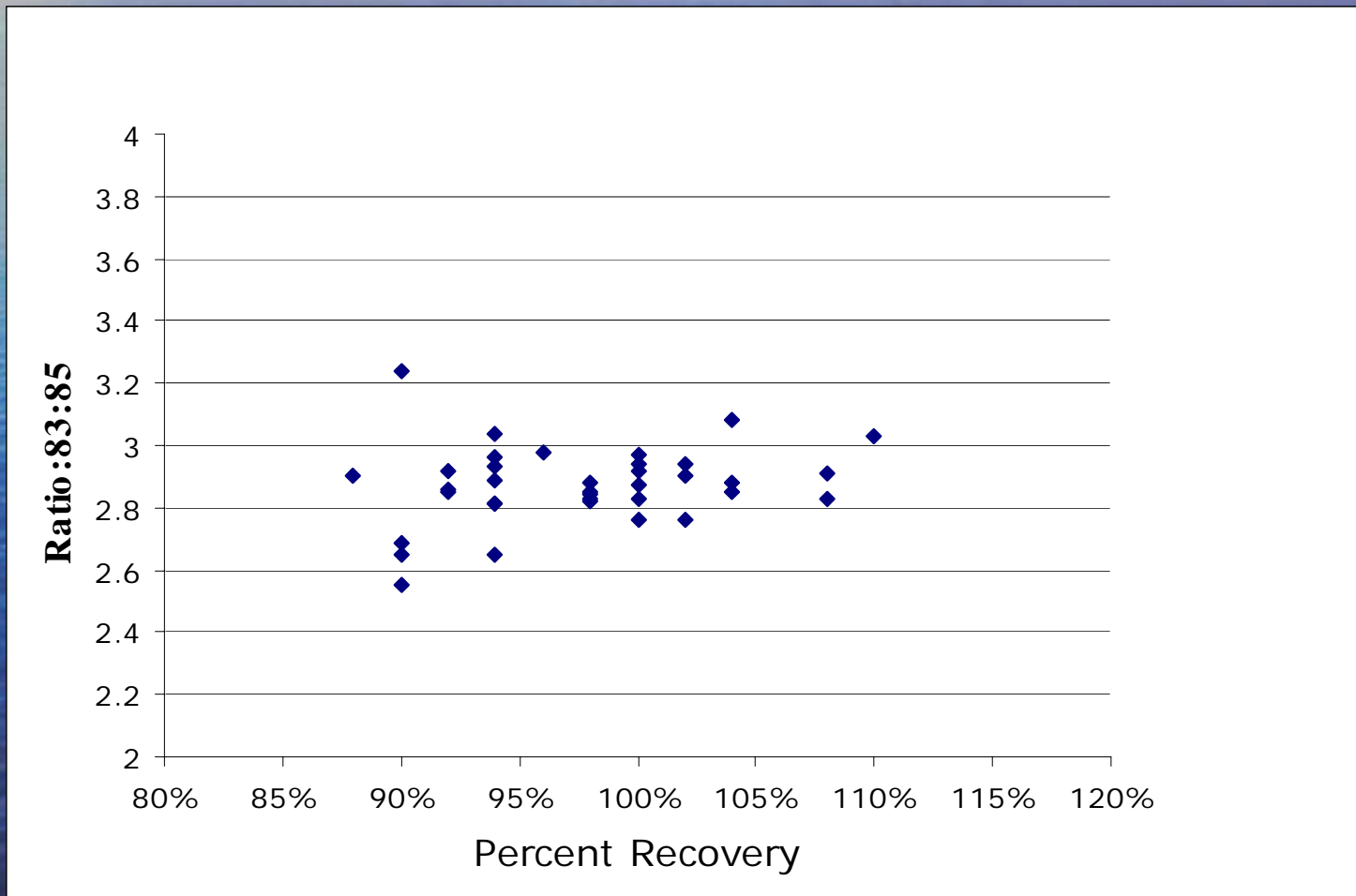


## Calibration and Performance Criteria (cont.)

- Initial and continuing calibration verification standards (ICV/CCV) :
  - from a second source
  - midpoint of the calibration range
  - within 15% of its true value
  - analyzed every ten samples



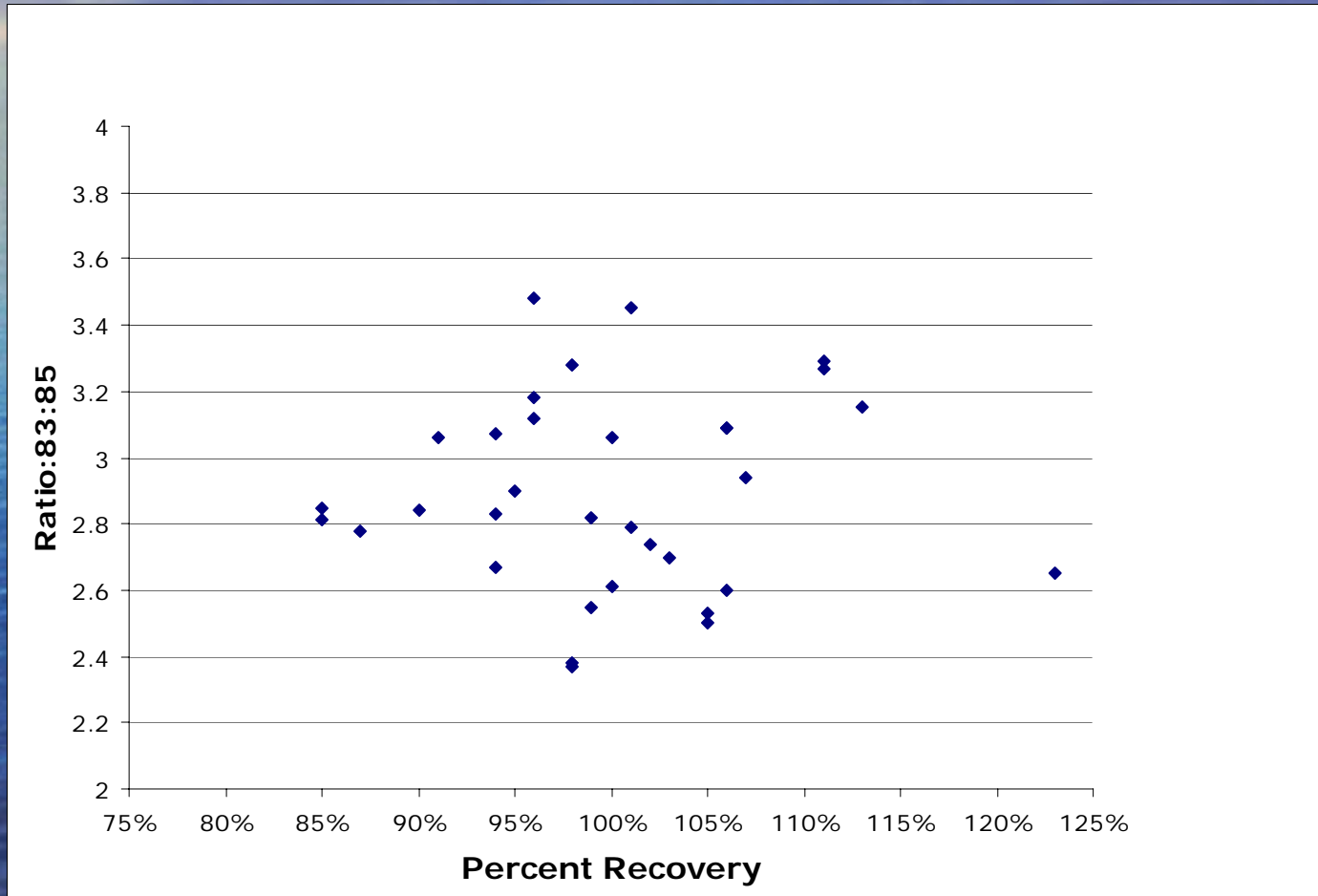
# ICV/CCV Recovery and Isotopic Ratios



## Calibration and Performance Criteria (cont.)

- MDL verification standard :
  - from the same source as the calibration
  - concentration is at the MDL
  - within 30% of its true value
  - analyzed every ten samples

# MDL Verification Recovery and Ratios





## Calibration and Performance Criteria (cont.)

- Instrument and continuing calibration blank (ICB/CCB):
  - Perchlorate concentration must be less than the MDL
  - Analyzed after each ICV and CCV

# Calibration and Performance Criteria (cont.)

- Prep blanks :
  - At least one per every 20 samples
  - Perchlorate concentration must be less than the MDL
- Laboratory Control Sample (LCS) :
  - At least one per every 20 samples
  - Perchlorate spike level at or near the PQL
  - Must be within 15% of true value



## Calibration and Performance Criteria (cont.)

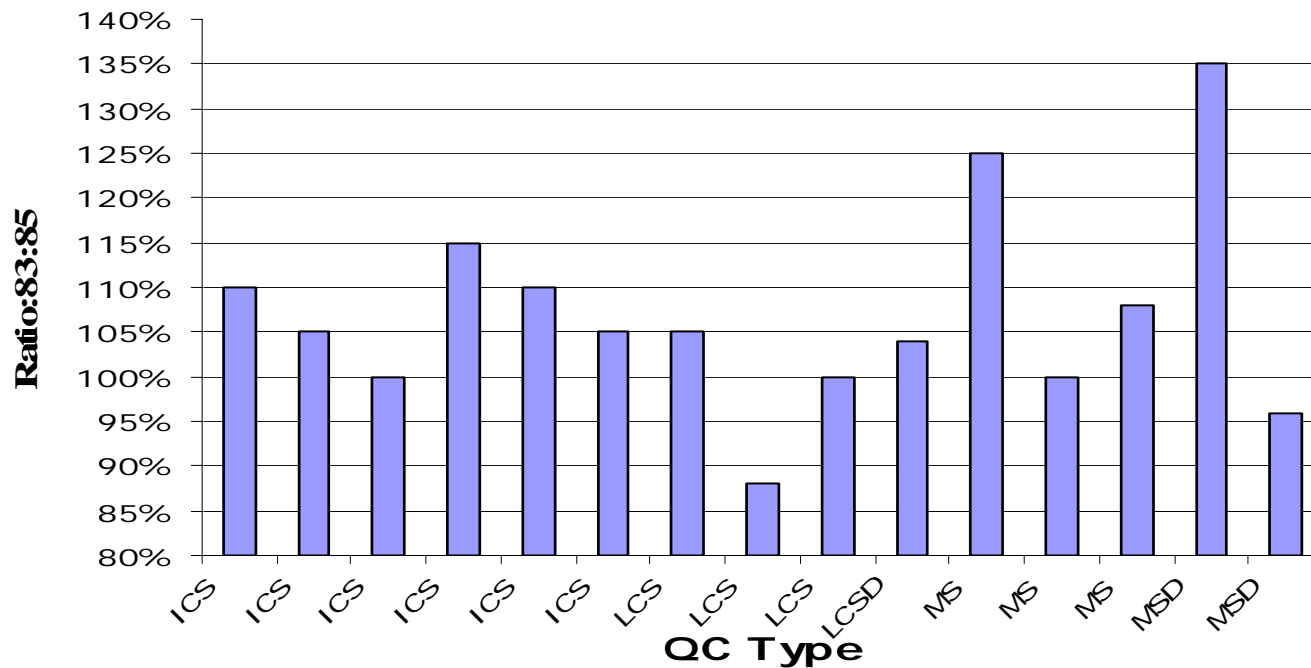
- Inteference Check Sample (ICS):
  - At least one every 20 samples
  - Contains 500 ppm each of sulfate, chloride, bicarbonate, and carbonate
  - Perchlorate spike level at or near the PQL
  - Must be within 20% of true value



## Calibration and Performance Criteria (cont.)

- Matrix Spike Samples (MS and MSD)
  - If sample volume allows, at least one pair every 20 samples
  - Perchlorate spike level at or near the PQL
  - Concentration must be within 25% of true value
  - RPD must be within 0 - 30%

# Extraction QC Recoveries



# Questions

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